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COMPARISON OF ANODIC DISCHARGABILITY OF LI-B ALLOY WITH PURE LI--ETC(U)
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**COMPARISON OF ANODIC DISCHARGABILITY
OF Li-B ALLOY WITH PURE Li IN
LiC₁₀ - PROPYLENE CARBONATE**

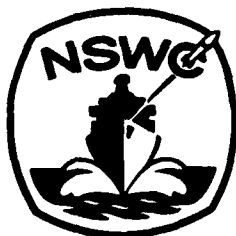
BY S. D. JAMES

RESEARCH AND TECHNOLOGY DEPARTMENT

APRIL 1981

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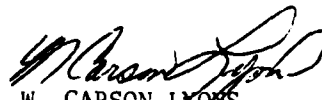
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FOREWORD

This report describes an experimental comparison of the anodic discharge behavior of Li-B alloy with pure Li in LiClO₄-propylene carbonate at 23°C. The results suggest that the alloy may find useful application in Li batteries when safety considerations permit the sacrifice of some coulombic capacity versus unsupported Li. We acknowledge the financial support of this work by the Independent Research Program of the Naval Surface Weapons Center and the help of Dr. Steven Dallek with Li-B ingot preparation.


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CHAPTER 1

INTRODUCTION

Safety problems in both the Li-SO₂ and the Li-SOCl₂ batteries have delayed their introduction into some advanced weapons systems. The present work seeks to reduce this problem by using a lithium-boron alloy in place of pure Li. The Li-rich, Li-B alloys were first prepared at NSWC in 1972¹. They showed impressive performance as high rate anodes in molten LiCl-KCl eutectic between 400 and 600°C². Many of the hazards of Li batteries have been associated with the melting of Li (at 180°C) in accidentally overheated cells. This problem should be considerably alleviated with Li-B anodes which retain their rigidity above 600°C. However, it is necessary to show that Li-B alloys can be discharged at room temperature at usefully high rates. In other words does the inert B component impede the discharge of the active Li? Hence the present work has characterized the anodic oxidation of Li-B alloy in LiClO₄-propylene carbonate (PC) solution at around 23°C as a function of anodic current density.

¹Wang, F. E., U. S. Patent 4,110,111, 29 Aug 1978.

²James, S. D. and DeVries, L.E., "Structure and Anodic Discharge Behavior of Li-B Alloys in the LiCl-KCl Eutectic Melt," J. Electrochem Soc., 123, 321, 1976.

CHAPTER 2

EXPERIMENTAL

PREPARATION OF THIN FILM TEST ELECTRODES

The Li-B alloy preparation has been described.³ In a Dry Room (below 3% R.H.) a 30g ingot was pressed between sheets of polypropylene to a circular pancake about 2x95 mm. From this cake a number of 10mm diameter (200mg) discs was cut out with a cork-borer. These discs were then pressed onto Stainless Steel (SS304) circular plates (32x1.6mm) which served as current collectors. The pressing operation (see Figure 1) produced thin film (0.1-0.2 mm) anodes that could undergo complete discharge of their free Li content in a reasonably short period of time. The Dake, Model 44-225 hydraulic press was capable of exerting 25 ton force. A key element in spreading thin alloy films over the 32 mm steel disc was the use of a deformable plastic disc above the Li-B alloy disc. When a rigid plastic (like polypropylene, PP) was used, 25 ton could not squeeze the alloy even to the steel periphery. However a deformable plastic like Conventional Polyethylene (CPE) or Nylon is itself squeezed out (irreversibly) and thereby effectively smears the alloy over the steel substrate. Three successive pressings using 5 ton force and a new plastic disc each time spread the alloy out well beyond the edge of the steel as shown in Figure 1. The result is a 0.1-0.2 mm thick anode film firmly stuck to the steel. Excess alloy film (about 100 mg) was pared away from the steel periphery and thermally analyzed³ for its free Li content. This provided a prediction of the coulombic capacity to be expected from this anode in its subsequent discharge. A final light pressing below rigid PP gave a glossy smooth finish to the anode surface. Similar test anodes of pure Li (Foote Mineral Co., 99.9%) were made to use as a standard by which to judge alloy discharge performance. With the much softer pure Li, use of a deformable plastic in the pressing was unnecessary.

DISCHARGE PROCEDURE

The Pyrex test-cell (Figure 2) contained 100 ml of electrolyte under dry argon. Its cap sealed via a Viton O-ring, pipe-joint. Stainless Steel (SS304) was extensively used in the cell as counter-electrode, test electrode support and 1/8" rod leads to all three electrodes. The test electrode disc was clamped horizontally, slightly recessed into its holder and about 15 mm below the bare steel counter electrode. Pure Li, plastered over the threaded tip of an 1/8" SS304 rod served as reference electrode. Heat-shrinkable polyolefine tubing masked off the steel surface of the reference electrode lead. All discharges used an electrolyte 0.96M, LiClO₄ ("lithium perchlorate anhydrous" from Foote Mineral Co.) in propylene carbonate (Eastman 7050). Electrolyte was prepared in the Dry Room and stored in Pyrex in the presence of Li ribbon. Discharge curves were recorded with constant anodic current applied to the test electrode at ambient temperature (23±1°C).

³Dallek, S., Ernst, D. W., and Larrick, B. F., "Thermal Analysis of Lithium-Boron Alloys," J. Electrochem. Soc., 126, 866, 1979.

CHAPTER 3

RESULTS AND DISCUSSION

Thermal measurements³ have been made on the free Li content of Li-rich, Li-B alloys such as those discharged in the present work. The data showed these alloys to consist of a refractory, Li-B compound (melting above 1000°C) having a stoichiometry in the vicinity of "Li₇B₆". Lithium in excess of "Li₇B₆", in free, elemental form is contained in fine pores permeating the refractory compound. At 500°C, most of this Li is available for anodic discharge at rates up to 8A cm⁻² at the Li potential². The compound behaves like a sponge and its liquid Li wicks to the alloy-melt interface to be discharged. At room temperature the free Li component is solid and rate capabilities of only mA cm⁻² are to be expected. The present work normally employed 80a/oLi (72w/oLi) alloy except in one case (Figure 4, 86 a/o Li).

Figures 3-6 compare the dischargability of Li-B alloy with that of pure Li at four anodic current densities ranging from 0.1 to 10 mA cm⁻². Where appropriate, anode voltage versus time is replotted as voltage versus percent utilization of the anode's total Li content (i.e. elemental Li plus Li chemically bound to B). Generally speaking the data show that over a wide range of current densities the alloy behaves comparably with pure Li, in terms of both voltage and coulombic capacity. The final plateau at around +5V (not included in Figures 5 and 6) is caused by anodic dissolution of the test electrode's steel substrate when Li ionization can no longer support the imposed current.

The relative complexity of the Li-B curves in Figures 3-5 is due to the two-phase nature of the alloy as previously shown in molten salt discharges^{2,4}. On the main V-plateau at the Li potential, free, elemental Li is anodically dissolving. When all the available (or accessible) free Li is used up, V rises to a value where tighter-bound Li can be anodized out of the decomposing compound. Subsequent inflections have been associated with the loss of further Li giving rise to other Li-B compound stoichiometries. As current-density rises in Figures 3 to 6 the Li-B curves gradually lose their complexity till, at 10mA cm⁻² there is no B-associated inflection at all. Apparently Li-B compound decomposition is too slow to generate sufficient Li to maintain the higher currents.

³See footnote 3 on page 9.

²See footnote 2 on page 7.

⁴DeVries, L. E., Jackson, L. D., and James, S. D., "Structure and Anodic Discharge Behavior of Li-B Alloys in the LiCl-KCl Eutectic Melt (II)," J. Electrochem Soc., 126, 993 (1979).

Figure 7 shows % utilization of the anode's total Li content to the first main inflection in the discharge curves of Figures 3-6. At 0.1 mA cm^{-2} , 100% of the pure Li is usable anodically. Above 1 mA cm^{-2} , utilization falls sharply. Discharge is now ended not by complete consumption of Li but by its superficial passivation. This takes the form of the sudden positive voltage excursion in Figures 3-6 terminating the anode's useful life. The nature of this polarization is presently under study. The Li-B alloy behaves similarly in Figure 7 except it utilizes somewhat less of its Li content. The value of 71% at 0.1 mA cm^{-2} probably reflects virtually complete consumption of the alloy's free Li since pure Li is 100% utilizable at this discharge rate. If an 80 a/o (72 w/o) Li-B alloy consisted of a mixture of Li_7B_6 compound plus elemental Li, this elemental Li would comprise 71% of the alloy's total Li. Thus Figure 7 supports the existence of " Li_7B_6 " in the Li-B system, agreeing with previous work in our laboratories^{3,5}. An unresolved conflict still exists between these three studies and the earlier molten salt discharges² which strongly suggested the presence of Li_2B . The single discharge made with the Li-rich, 86 a/o alloy was at too high a rate (1 mA cm^{-2}) to yield stoichiometric information but its 10% higher Li-utilization (versus the 80 a/o) is consistent with the presence of " Li_7B_6 " (free Li comprises 81% of total Li in 86 a/o alloy containing $\text{Li}_7\text{B}_6 + \text{Li}$).

³ See footnote 3 on page 9.

⁵ Kilroy, W. P. and Angres, I., "The Extraction and Determination of Free Lithium in Li-B Alloys," J. Less-Common Met., 63, 123, 1979.

CHAPTER 4

CONCLUSIONS

1. Over a wide range of anodic current density, Li-B alloy behaves comparably with pure Li in terms of the voltage and coulombic capacity of its discharge in PC-LiClO₄.
2. Extrapolation of Li utilizability to low discharge rate suggests that the Li-B alloy is a mixture of pure Li and a Li-B compound whose stoichiometry is in the vicinity of "Li₇B₆".
3. Table 1 compares the discharge capacities of Li-B alloy and pure Li at 1mA cm⁻² in propylene carbonate. The alloy has only 47 and 65% of the capacity of pure Li on a gravimetric and volumetric basis respectively. However, if Li is supported in a rigid, refractory matrix like Li-B or Ni Feltmetal (FM) it is likely to be safer in the event that a battery overheats and its Li component melts. On a gravimetric and volumetric basis the alloy has 157 and 81% of the capacity of Li-FM. Furthermore reference (2) showed that, between 400 and 600°C, Li escapes significantly more slowly from Li-B than from Li-FM. Thus the Li-B alloy may find useful application where safety considerations permit the sacrifice of some coulombic capacity versus unsupported Li.

TABLE 1 USEFUL COULOMBIC CAPACITIES¹ AT THE Li POTENTIAL
DELIVERED BY Li-B², Li-FM³ AND PURE Li

	<u>Coulomb/cc, anode</u>	<u>Coulomb/g, anode</u>
Li-B	4465	6177
Li-FM	5500	3900
Pure Li	6905	12930

1. Coulomb/cc or g of (Li+B) for alloy and (Li+Ni) for Li-FM. Pure Li and Li-B discharged (as thin films 0.1-0.2mm) in 0.96M LiClO₄-PC, 22°C. Capacity calculated at first main inflection of 1mA cm⁻² discharge curves.
2. Li content: 0.80 atom fraction; 0.72 mass fraction.
3. Li in 82% porous, 8x6mm plugs of Ni Feltmetal discharged at 500°C (reference 2).

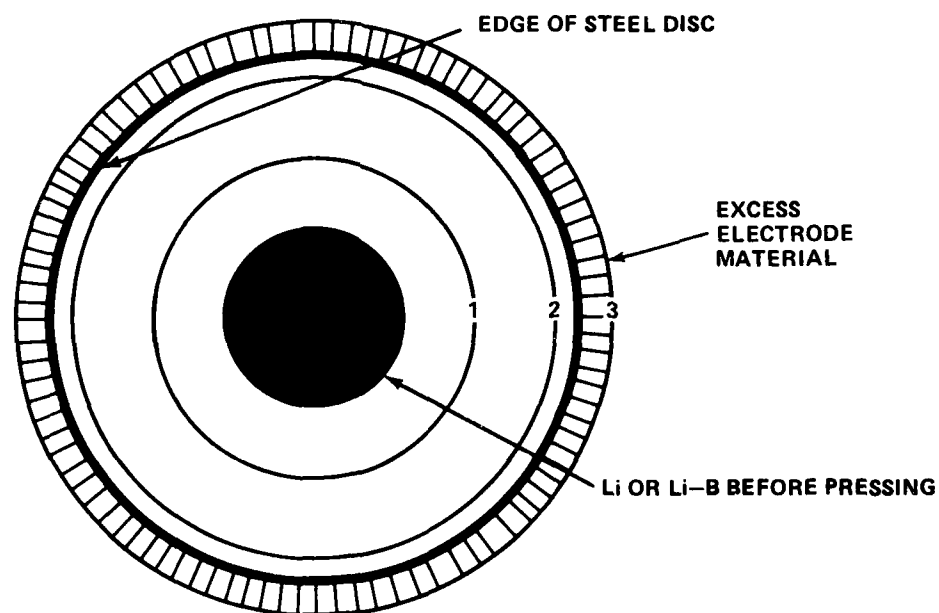
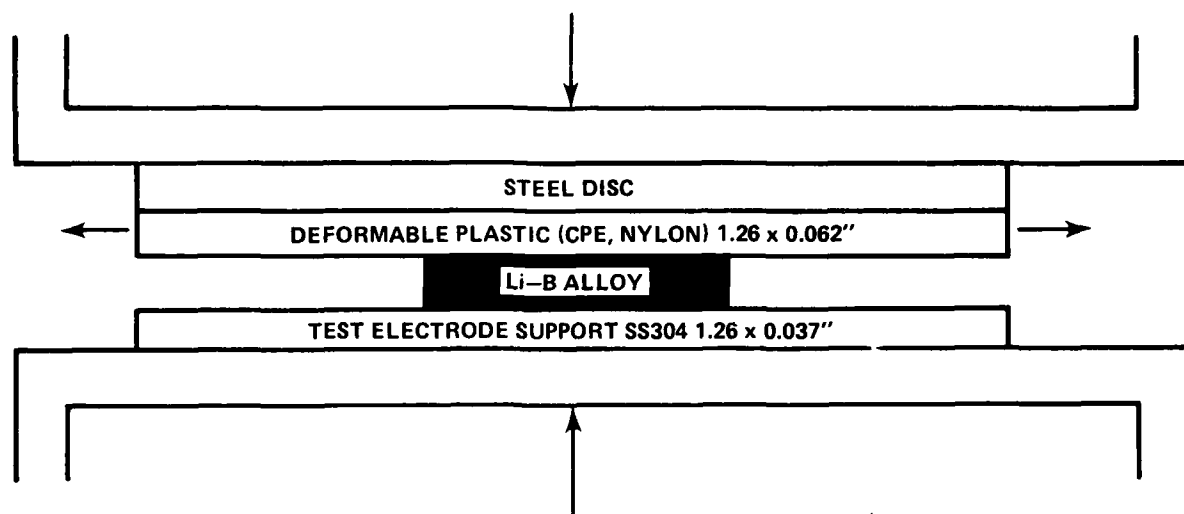


FIGURE 1 PREPARATION OF THIN FILM TEST ELECTRODES BY 3-STAGE PRESSING

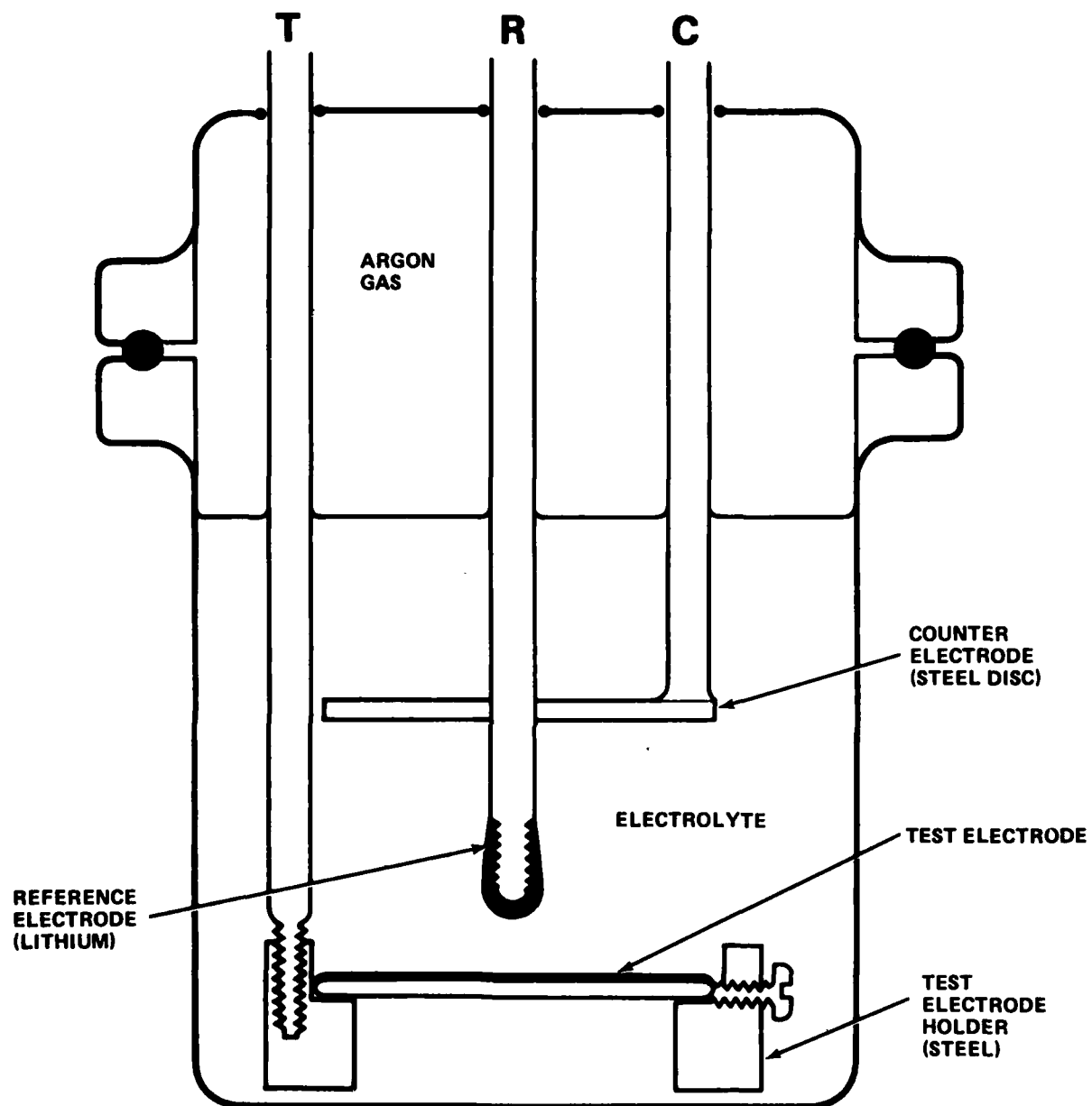


FIGURE 2 DISCHARGE CELL

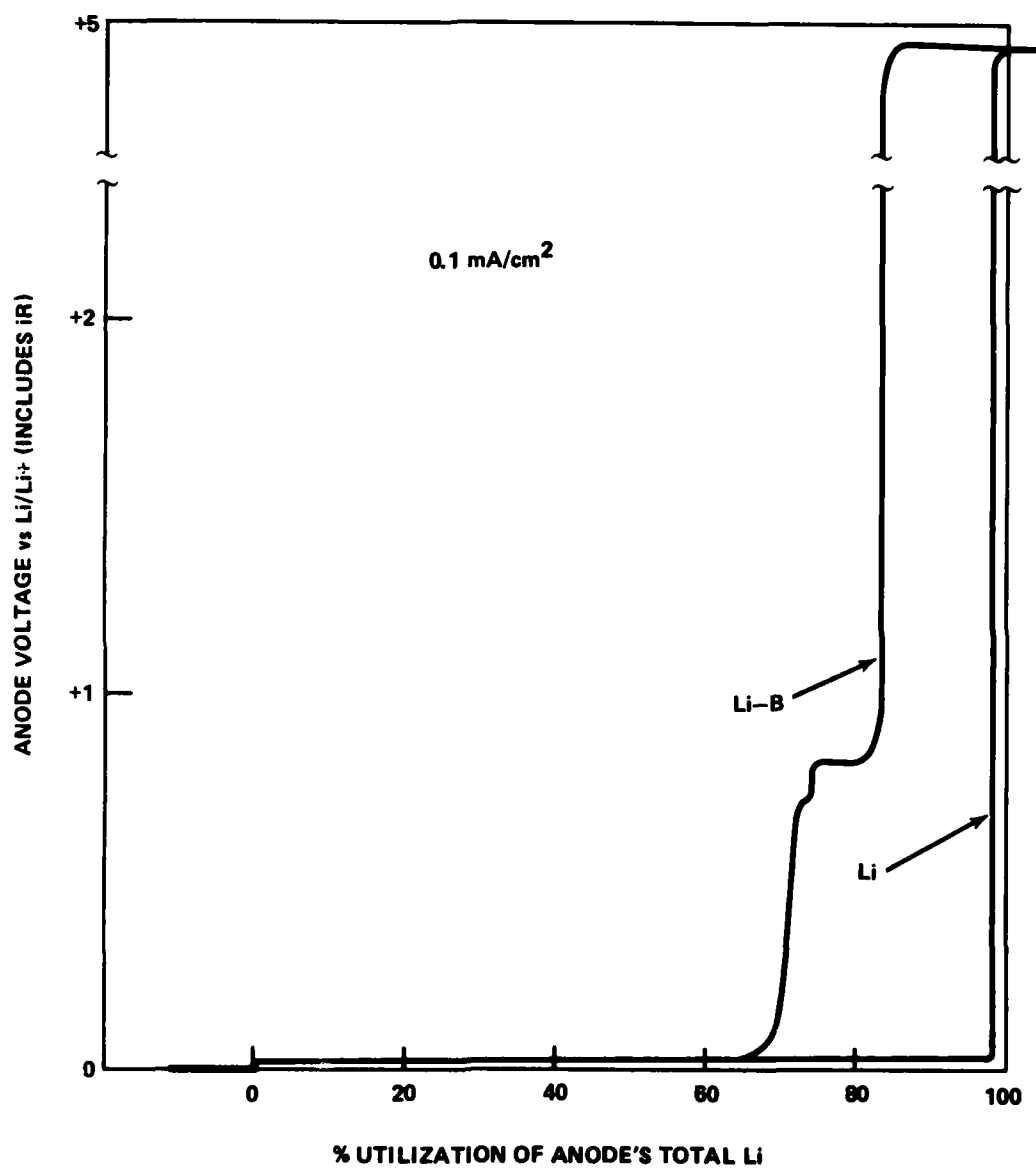


FIGURE 3 ANODIC DISCHARGE OF LI AND LI-B (80 a/o LI) IN 0.96 M LiClO₄-PC AT 0.1 mA cm⁻², 23°C

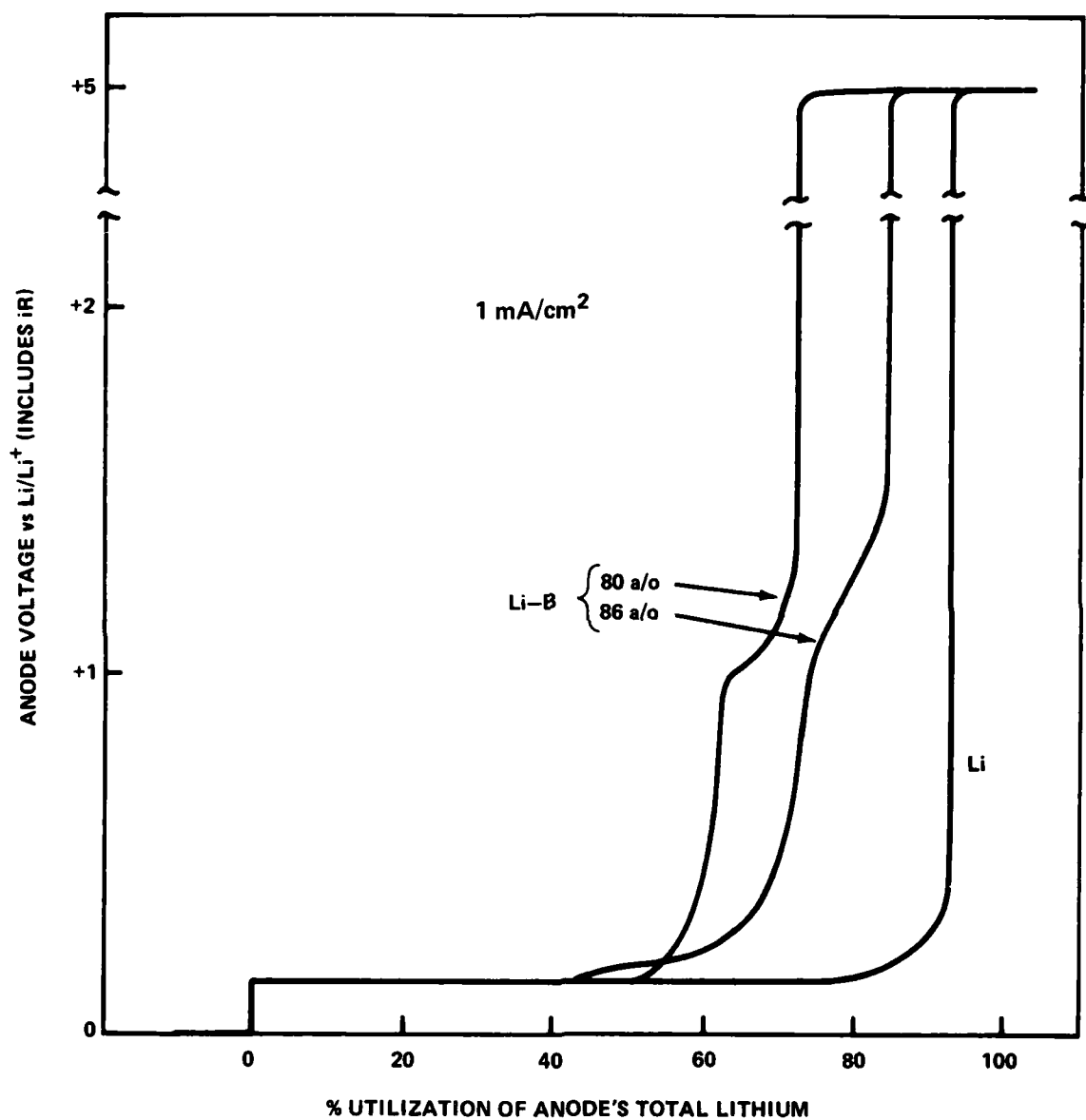
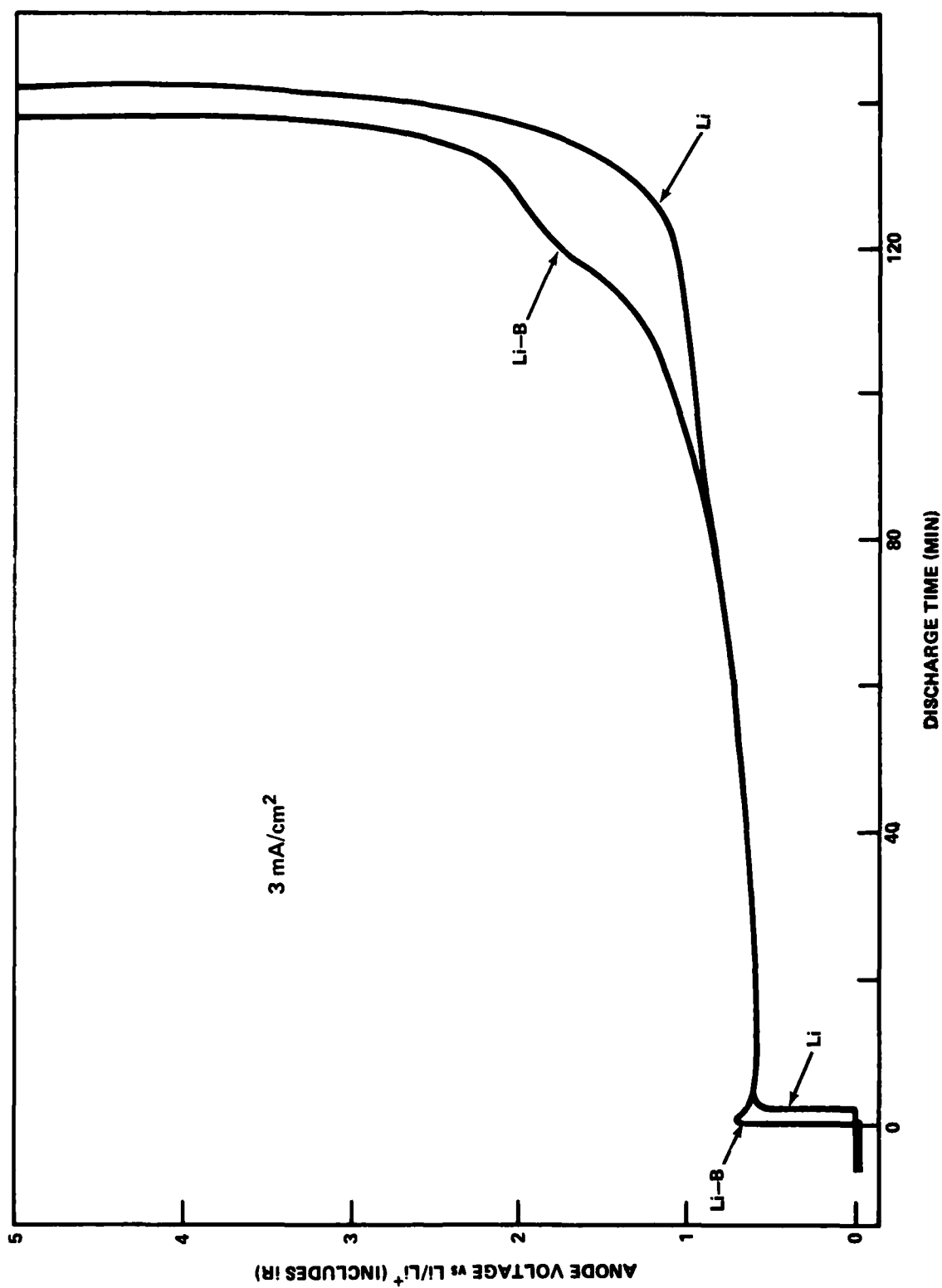


FIGURE 4 ANODIC DISCHARGE OF Li AND Li-B (80 and 86 a/o Li) IN $0.96 \text{ M LiClO}_4\text{-PC}$ AT 1 mA cm^{-2} , 23°C

FIGURE 5 ANODIC DISCHARGE OF LI AND LI-B (80 a/o) IN 0.08 M LiClO₄-PC AT 3 mA cm⁻², 23°C

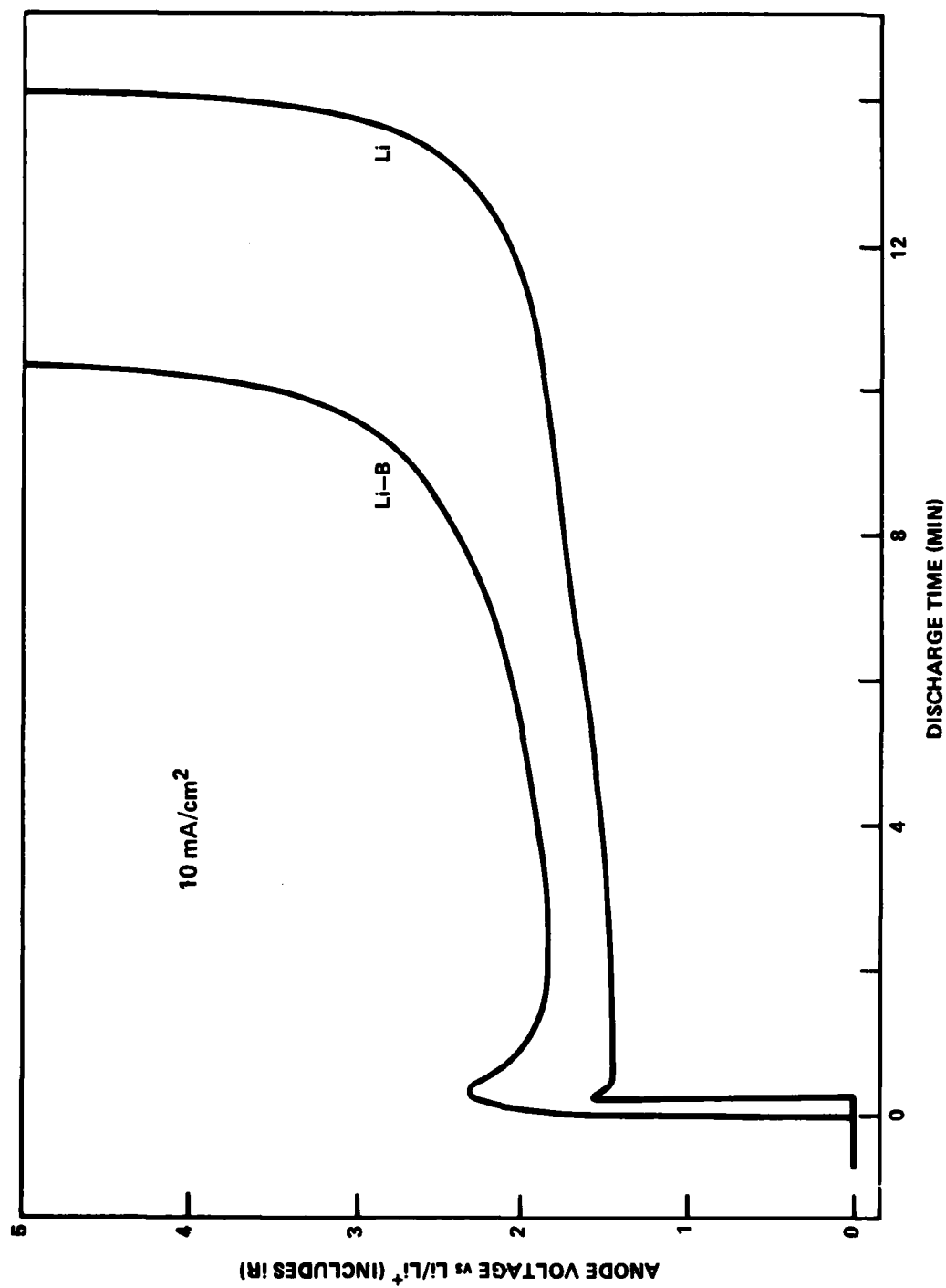


FIGURE 6 ANODIC DISCHARGE OF Li AND Li-B (80 a/o) IN 0.96 M LiClO₄-PC AT 10 mA cm⁻², 23°C

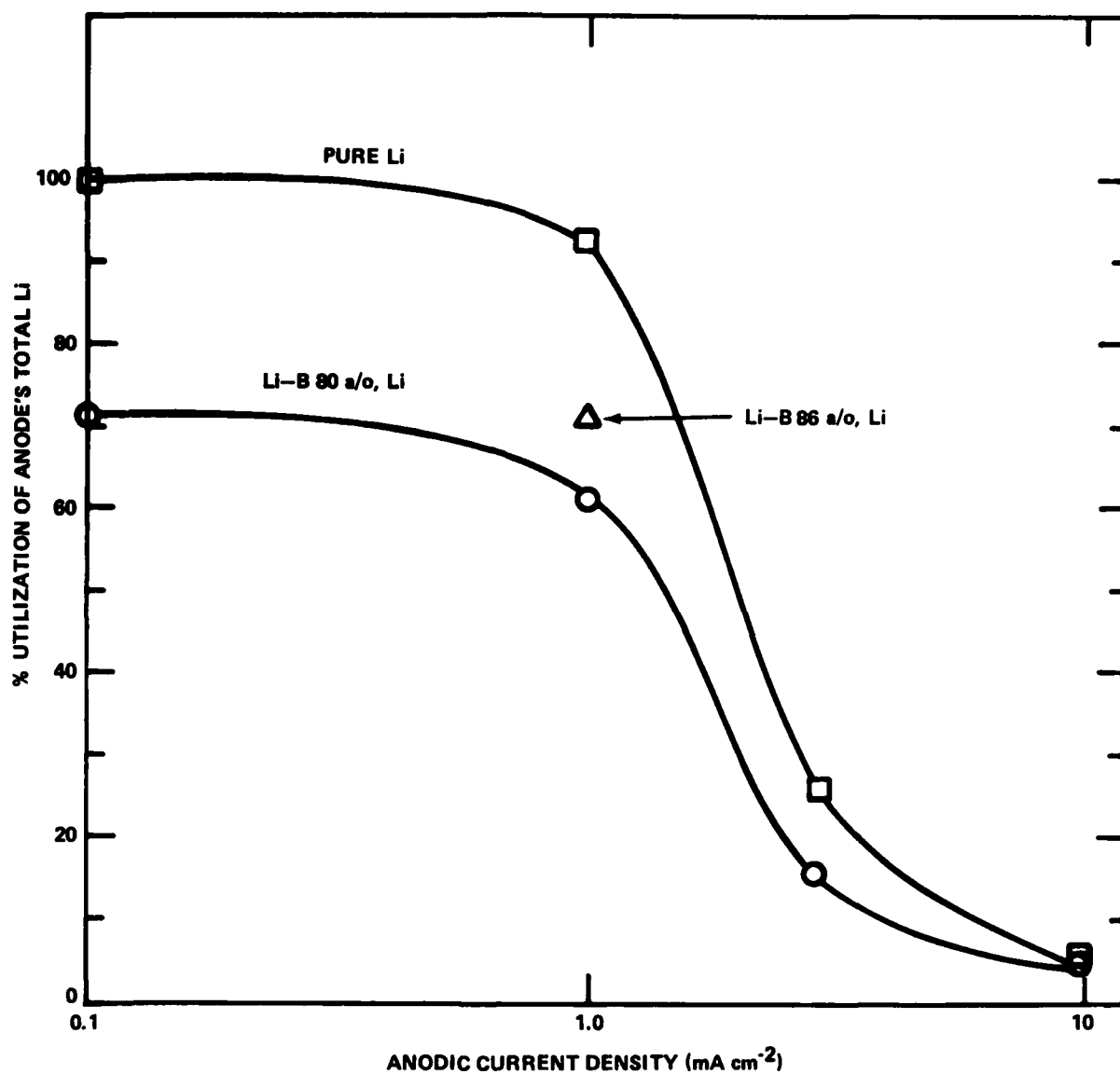


FIGURE 7 % ANODIC UTILIZATION OF THE TOTAL LI (ELEMENTAL PLUS CHEMICALLY BOUND) IN 0.02mm THICK FILMS OF LI AND LI-B (CALCULATED AT THE FIRST INFLECTION OF DISCHARGE CURVES IN 0.96 M LiClO₄-PC, 23°C)

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